

FORM PTO-1350 (REV. 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER Bayer 10,219-WCG
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (If known, see 37 CFR 1.5) 09/913947
INTERNATIONAL APPLICATION NO. PCT/EP00/01206	INTERNATIONAL FILING DATE 14 February 2000 (14.02.00)	PRIORITY DATE CLAIMED 24 February 1999 (24.02.99)	
TITLE OF INVENTION METHOD AND DEVICE FOR PRODUCING ELASTANE THREADS FROM RECYCLING MATERIAL			
APPLICANT(S) FOR DO/EO/US Ulrich REINEHR, Tilo SEHM, Wolfgang ANDERHEGGEN, Toni HERBERTZ and Helmut JUDAT			
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:			
<p>1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))</p> <p>a. <input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).</p> <p>a. <input checked="" type="checkbox"/> is attached hereto.</p> <p>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</p> <p>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))</p> <p>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input type="checkbox"/> have been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p>d. <input type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).</p> <p>9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</p> <p>Items 11 to 20 below concern document(s) or information included:</p> <p>11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input checked="" type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A FIRST preliminary amendment.</p> <p>14. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input checked="" type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</p> <p>19. <input checked="" type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input checked="" type="checkbox"/> Other items or information:</p> <p>1) Copy of published application WO 00/50673, with International Search Report attached.</p> <p>2) Copy of International Preliminary Examination Report</p> <p>3) Copy of Original Request</p>			

21. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

Neither international preliminary examination fee (37 CFR 1.482)
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO
and International Search Report not prepared by the EPO or JPO. \$1000.00International preliminary examination fee (37 CFR 1.482) not paid to
USPTO but International Search Report prepared by the EPO or JPO \$860.00International preliminary examination fee (37 CFR 1.482) not paid to USPTO
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO \$710.00International preliminary examination fee (37 CFR 1.482) paid to USPTO
but all claims did not satisfy provisions of PCT Article 33(1)-(4) \$690.00International preliminary examination fee (37 CFR 1.482) paid to USPTO
and all claims satisfied provisions of PCT Article 33(1)-(4) \$100.00

ENTER APPROPRIATE BASIC FEE AMOUNT =

CALCULATIONS PTO USE ONLY

\$ 860.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$

CLAIMS NUMBER FILED NUMBER EXTRA RATE \$

Total claims 33 -20 = 13 x \$18.00 \$ 234.00

Independent claims 3 -3 = 0 x \$80.00 \$

MULTIPLE DEPENDENT CLAIM(S) (if applicable) + \$270.00 \$

TOTAL OF ABOVE CALCULATIONS = \$

☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above
are reduced by 1/2. \$

SUBTOTAL = \$ 1,094.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)). \$

TOTAL NATIONAL FEE = \$ 1,094.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property + \$

TOTAL FEES ENCLOSED = \$ 1,094.00

Amount to be
refunded: \$

charged: \$

- a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.
- b. ☒ Please charge my Deposit Account No. 14-1263 in the amount of \$ 1,094.00 to cover the above fees.
A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any
overpayment to Deposit Account No. 14-1263. A duplicate copy of this sheet is enclosed.
- d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card
information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR
1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

William C. Gerstenzang
Norris, McLaughlin & Marcus
220 East 42nd Street
New York, N.Y. 10017
Tel.# 212-808-0700
Fax# 212-808-0844

SIGNATURE

William C. Gerstenzang

NAME

27,552

REGISTRATION NUMBER

09/913947

JC05 Rec'd PCT/PTO 20 AUG 2001

Attorney Docket No.: Bayer 10,219-WCG
FAS 13-US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s) : Ulrich REINEHR, Tilo SEHM, Wolfgang
ANDERHEGGEN, Toni HERBERTZ and Helmut JUDAT

PCT Application No.: PCT/EP00/01206

Serial No. : To Be Assigned

Filed : Herewith

For : METHOD AND DEVICE FOR PRODUCING ELASTANE
THREADS FROM RECYCLING MATERIAL

Art Unit : To Be Assigned

Examiner : To Be Assigned

August 20, 2001

BOX PCT
Hon. Assistant Commissioner For Patents
Washington, D.C. 20231

PRELIMINARY AMENDMENT

Sir:

In advance of prosecution, kindly amend the above-identified application as follows and consider the following remarks:

IN THE DRAWINGS

Please consider and approve the accompanying corrected copies of Figs. 1-3.

IN THE CLAIMS

Please cancel the previous version of the following claims and replace them with the following rewritten version. A marked up copy showing the amendment since the previous version is annexed as separate pages.

09913947-000001

Claim 1 (amended). Process for producing elastane fibres by the dry spinning process or the wet spinning process using spinning solutions of elastane recycling material, optionally with the addition of fresh elastane solution, wherein

- a) the waste obtained from elastane fibres, is cut up to a cut length of at least 0.1 mm,
- b) the cut elastane fibers and 0.1 to 2 wt.%, based on the content of solid elastane, of a secondary aliphatic amine are introduced into the spinning solvent,
- c) the mixture of cut elastane fibers, spinning solvent and secondary aliphatic amine is heated at a temperature of 60°C to 150°C, with homogenisation,
- d) the resulting homogeneous elastane spinning solution is prefiltered,
- e) optionally with the addition of fresh elastane solution, the elastane spinning solution is mixed at 70°C to 140°C with a fresh elastane solution, in any mixing ratio and heating is continued,
- f) the resulting spinning solution is cooled to a temperature of at not more than 70°C, and filtered once more,

- g) the finished spinning solution is optionally remixed, and subsequently spun into elastane fibres by the dry spinning process or the wet spinning process.

Claim 2 (amended). Process according to claim 1, wherein the elastane waste comprises both elastanes based on polyether and elastanes based on polyester, or wastes of mixed polyether- and polyester-containing elastanes in any mixing ratio.

Claim 3 (amended). Process according to claim 1 wherein the secondary aliphatic amine used is diethylamine (DEA), in a quantity of 0.3 to 1 wt.%, based on solid elastane.

Claim 4 (amended). Process according to Claim 1, wherein the concentration of the finished spinning solution in step h) is from 22 to 45 wt. %.

Claim 5 (amended). Process according to Claim 1, wherein dimethylacetamide is used as the spinning solvent.

Claim 6 (amended). Process according to Claim 1, wherein the elastane spinning solution is mixed with a fresh elastane solution in step f) for a period of 5 to 60 minutes.

Claim 7 (amended). Process according to Claim 1, wherein the total mixing time in steps b) and c) together is at least 10 minutes.

Claim 8 (amended). Process according to Claim 1, wherein the solution temperature in step c) is from 80°C to 120°C.

Claim 9 (amended). Process according to Claim 1, wherein in the case where a mixture of waste elastane solution and fresh elastane solution is used, the required addition of secondary aliphatic amine to the fresh elastane solution is carried out not in step b) but in step f) and in the form of a stock batch of secondary aliphatic amine and fresh elastane solution.

Claim 10 (amended). Process according to Claim 1, wherein the recycling spinning solution and the fresh solution are preferably mixed together in a static mixer, at 70°C to 140°C for a period of 5 to 30 minutes and the spinning solution is subsequently cooled to 50°C to 70°C.

Claim 11 (amended). Process according to Claim 1, wherein the solids content of the recycling spinning solution in proportion to the solids concentration of the total spinning solution comprising recycling spinning solution and fresh elastane solution is at least 10 wt.%.

Claim 12 (amended). Process according to Claim 1, wherein the spinning of the spinning solutions by the wet spinning process is effected from spinnerets having a nozzle hole diameter of 0.10 to 0.3 mm.

Claim 13 (amended). Process according to Claim 1, wherein the elastane fibers in step a) is introduced in portions into the vortex created by an agitated disperser in the previously prepared spinning solvent.

Claim 14 (amended). Elastane fibres obtained from a process according to Claim 1 with a titre of up to 10,000 dtex, and fibre strengths of 0.5 to 0.95 cN/dtex and elongation at tear of 500 to 750%.

Claim 15 (amended). Device for carrying out the process of Claim 1, comprising at least one heatable mixing tank with an inlet for solid material, a mixer and a dispersing unit, a mixing zone with static mixing elements, connected downstream of the mixing tank, a cooling zone with mixing elements and a filtering unit for subsequently filtering the prepared elastane spinning solution.

Claim 16 (amended). Device according to claim 15, wherein the dispersing unit consists of at least one agitated disperser.

Claim 17 (amended). Device according to claim 16, at least one agitated disperser is equipped with screw-type segmented appliances.

Claim 18 (amended). Device according to claim 16, wherein the at least one agitated disperser is equipped with screw-type segmented appliances selected from the group consisting of suction cutters, webbed rings, kneading spirals and multiple current appliances.

Claim 19 (amended). Device according to Claim 15, wherein the mixer is an anchor mixer and the mixing tank is provided with an additional cleaning device for the internal fittings of the mixer.

Please add the following:

--Claim 20. The process of Claim 1, wherein said cut length is at least 1 mn.

Claim 21. The process of Claim 20, wherein said cut length is from 2 to 10 mn.

Claim 22. The process of Claim of 21, wherein said cut length is 3 to 5 mn.

Claim 23. The process of Claim of 1, wherein the cooling of step f) is to a temperature of 50°C to 70°C.

Claim 24. The process of Claim of 1, wherein the optional remixing of step g) is by stirring and degassing.

Claim 25. The process of Claim of 3, wherein said quantity of diethylamine is 0.5 to 0.8 wt %.

Claim 26. The process of Claim of 4, wherein said concentration is from 30 to 40 wt %.

Claim 27. The process of Claim of 7, wherein said total mixing time is from 60 to 150 minutes.

Claim 28. The process of Claim of 26, wherein said total mixing time is 90 to 120 minutes.

Claim 29. The process of Claim of 9, wherein said secondary aliphatic amine is diethylamine.

Claim 30. The process of Claim of 10, wherein said temperature is 100°C to 120°C.

Claim 31. The process of Claim 12, wherein said hole diameter is 0.1 to 0.2 mm.

Claim 32. The process of Claim 13, wherein said elastane fibers is introduced via a metering or weighing device at a rate of 1 to 10 kg/minute.

Claim 33. The device of Claim 16, wherein said dispersing unit consists of two agitated dispersing.--

REMARKS

This Preliminary Amendment is being filed to eliminate multiple dependency, conform the claims to conventional format and to enter the Substitute Sheets of Drawings that were filed under Rule 26 during the international stage.

Favorable action is respectfully solicited.

CONDITIONAL PETITION FOR EXTENSION OF TIME

If any extension of time for this amendment is required, applicant requests that this be considered a petition therefore. Please charge the required petition fee to Deposit Account No. 14-1263.

ADDITIONAL FEE

Please charge any insufficiency of fee or credit any excess to Deposit Account No. 14-1263.

Respectfully submitted,

NORRIS, McLAUGHLIN & MARCUS, P.A.

By 

William C. Gerstenzang
Reg. No. 27,552

220 East 42nd Street - 30th Floor
New York, New York 10017
(212) 808-0700

I hereby certify that this paper is being deposited with the United States Postal Service as Express Mail, Label No. EL867733455US to: BOX PCT, The Hon. Commissioner of Patents, Washington, D.C. 20231 on August 20, 2001.

Norris McLaughlin & Marcus, P.A.

By: 

Date: 8/20/01

3/PRT3

09/913947

JC05 Rec'd PCT/PTC

20 AUG 2001

**MARKED-UP COPY OF AMENDED CLAIM,
SHOWING CHANGES RELATIVE TO PREVIOUS VERSION**

Claim 1 (amended). Process for producing elastane fibres by the dry spinning process or the wet spinning process using spinning solutions of elastane recycling material, optionally with the addition of fresh elastane solution, [characterised in that] wherein

- a) the waste obtained from elastane [material, in particular from elastane] fibres, is cut up[, in particular] to a cut length of at least 0.1 mm, [preferably to a cut length of at least 1 mm, particularly preferably to a cut length of 2 to 10 mm, most preferably to a cut length of 3 to 5 mm,]
- b) the cut elastane [material] fibers and 0.1 to 2 wt.%, based on the content of solid elastane, of a secondary aliphatic amine are introduced into the spinning solvent,
- c) the mixture of cut elastane [material] fibers, spinning solvent and secondary aliphatic amine is heated at a temperature of 60°C to 150°C, with homogenisation,
- d) the resulting homogeneous elastane spinning solution is prefiltered,
- e) optionally with the addition of fresh elastane solution, the elastane spinning solution is mixed at 70°C to 140°C with a fresh elastane solution, [preferably having the same solids concentration,] in any mixing ratio and heating is continued,

- f) the resulting spinning solution is cooled to a temperature of at not more than 70°C, [preferably from 50°C to 70°C,] and filtered once more,
- g) the finished spinning solution is optionally remixed, [for example, by stirring, degassed] and subsequently spun into elastane fibres by the dry spinning process or the wet spinning process.

Claim 2 (amended). Process according to claim 1, [characterised in that] wherein the elastane waste [consists of] comprises both elastanes based on polyether and elastanes based on polyester, or [of] wastes of mixed polyether- and polyester-containing elastanes in any mixing ratio.

Claim 3 (amended). Process according to claim 1 [or 2, characterised in that] wherein the secondary aliphatic amine used is diethylamine (DEA), in a quantity [preferably] of 0.3 to 1 wt.%, [in particular preferably of 0.5 to 0.8 wt.%,] based on solid elastane.

Claim 4 (amended). Process according to [claims 1 to 3, characterised in that] Claim 1, wherein the concentration of the finished spinning solution in step h) is from 22 to 45 wt.% [, preferably from 30 to 40 wt.%].

Claim 5 (amended). Process according to [claims 1 to 4, characterised in that] Claim 1, wherein dimethylacetamide is used as the spinning solvent.

09913947-082601

Claim 6 (amended). Process according to [claims 1 to 5, characterised in that] Claim 1, wherein the elastane spinning solution is mixed with a fresh elastane solution in step f) for a period of 5 to 60 minutes.

Claim 7 (amended). Process according to [claims 1 to 6, characterised in that] Claim 1, wherein the total mixing time in steps b) and c) together is at least 10 minutes[, preferably from 60 to 150 minutes, particularly preferably 90 to 120 minutes].

Claim 8 (amended). Process according to [claims 1 to 7, characterised in that] Claim 1, wherein the solution temperature in step c) is from 80°C to 120°C.

Claim 9 (amended). Process according to [claims 1 to 8, characterised in that] Claim 1, wherein in the case where a mixture of waste elastane solution and fresh elastane solution is used, the required addition of secondary aliphatic amine[, in particular of diethylamine,] to the fresh elastane solution is carried out not in step b) but in step f) and in the form of a stock batch of secondary aliphatic amine and fresh elastane solution.

Claim 10 (amended). Process according to [claims 1 to 9, characterised in that] Claim 1, wherein the recycling spinning solution and the fresh solution are preferably mixed together in a static mixer, [preferably] at 70°C to 140°C[, in particular at 100°C to 120°C,] for a period of 5 to 30 minutes and the spinning solution is subsequently cooled to 50°C to 70°C.

0943947-082004

Claim 11 (amended). Process according to [claims 1 to 10, characterised in that] Claim 1, wherein the solids content of the recycling spinning solution in proportion to the solids concentration of the total spinning solution comprising recycling spinning solution and fresh elastane solution is at least 10 wt.% [, preferably at least 20 wt.%].

Claim 12 (amended). Process according to [claims 1 to 11, characterised in that] Claim 1, wherein the spinning of the spinning solutions by the wet spinning process is effected from spinnerets having a nozzle hole diameter of 0.10 to 0.3 mm[, preferably of 0.1 to 0.2 mm].

Claim 13 (amended). Process according to [claims 1 to 12, characterised in that] Claim 1, wherein the elastane [material] fibers in step a) is introduced in portions[, in particular via a metering/weighing device, preferably at a rhythm of 1 to 10 kg/minute, particularly preferably 3 to 5 kg/minute,] into the vortex created by an agitated disperser in the previously prepared spinning solvent.

Claim 14 (amended). Elastane fibres obtained from a process according to Claim 1 [one of claims 1 to 13, in particular] with a titre of up to 10,000 dtex, [characterised in that their] and fibre strengths [is] of 0.5 to 0.95 cN/dtex[, preferably 0.7 to 0.9 cN/dtex,] and [the] elongation at tear [is] of 500 to 750%[, preferably 550 to 700%].

Claim 15 (amended). Device for carrying out the process [according to one of claims 1 to 13, consisting] of Claim 1, comprising at least [of a] one heatable

mixing tank [1] with an inlet [2, 6] for solid material, a mixer [3] and a dispersing unit [4, 5, of], a mixing zone [7] with static mixing elements, connected downstream of the mixing tank [1, of], a cooling zone [11] with mixing elements and [of] a filtering unit [12] for subsequently filtering the prepared elastane spinning solution.

Claim 16 (amended). Device according to claim 15, [characterised in that] wherein the dispersing unit [4, 5,] consists of at least one[, in particular two,] agitated disperser[s].

Claim 17 (amended). Device according to claim 16, [characterised in that the] at least one agitated disperser[(s) 4 is/are] is equipped with screw-type segmented appliances [5].

Claim 18 (amended). Device according to claim 16, [characterised in that] wherein the at least one agitated disperser[(s) 4 is/are] is equipped with screw-type segmented appliances [5] selected from the group consisting of suction cutters, webbed rings, kneading spirals and multiple current appliances.

Claim 19 (amended). Device according to [one of claims 14 to 17, characterised in that] Claim 15, wherein the mixer [3] is an anchor mixer and the mixing tank [1] is provided with an additional cleaning device for the internal fittings of the mixer [3].

09/913947

1999 JUL 14 11:00 AM '99

Attorney Docket No.: Bayer 10,219-WCG
FAS 13-US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s) : Ulrich REINEHR, Tilo SEHM, Wolfgang ANDERHEGGEN, Toni
HERBERTZ and Helmut JUDAT

PCT Application No.: PCT/EP00/01206

Serial No. : To Be Assigned

Filed : Herewith

For : METHOD AND DEVICE FOR PRODUCING ELASTANE
THREADS FROM RECYCLING MATERIAL

Art Unit : To Be Assigned

Examiner : To Be Assigned

August 20, 2001

The Assistant Commissioner
for Patents
Washington, D.C. 20231

Sir:

LETTER TO THE OFFICIAL DRAFTSMAN

Submitted herewith for entry and approval is three (3) sheets of formal drawings in the
above-referenced application.

Respectfully submitted,
NORRIS, McLAUGHLIN & MARCUS, P.A.

By William C. Gerstenzang
William C. Gerstenzang
Reg. No. 27,552

WCG:gb
Enc. - Formal Drawings (3 sheets)

220 East 42nd Street
30th Floor
New York, New York 10017
Phone: (212) 808-0700
Fax: (212) 808-0844

I hereby certify that this correspondence is being deposited with the
United States Postal Services as Express Mail in an envelope
addressed to The Hon. Commissioner for Patents, Washington,
D.C. 20231 on August 20, 2001.

NORRIS, McLAUGHLIN & MARCUS, P.A.

By William C. Gerstenzang

Date 8/20/01

09/913947-000001

- 1 / 3 -

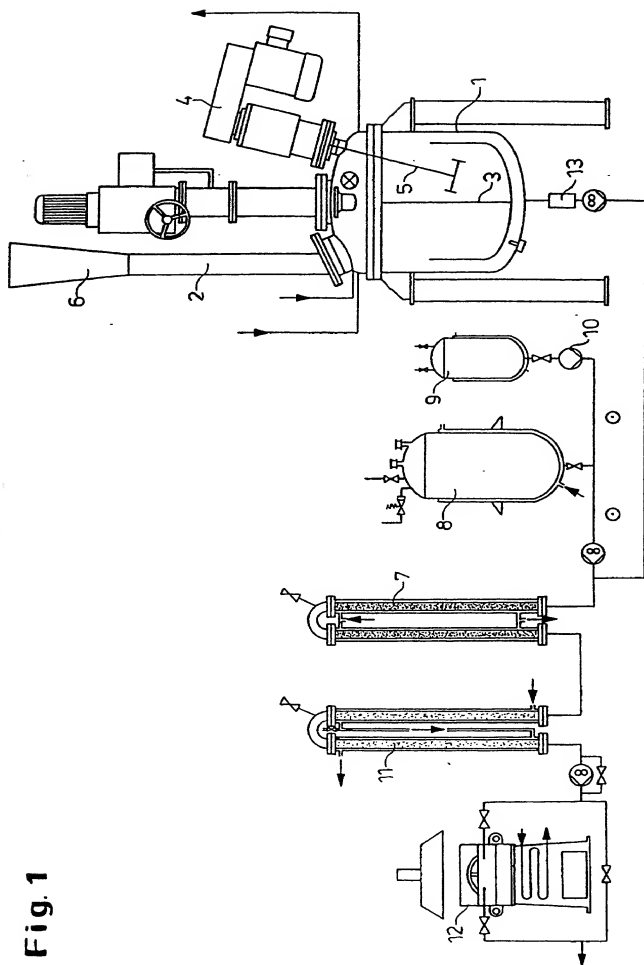


Fig. 1

SCANNED 24

Fig. 2

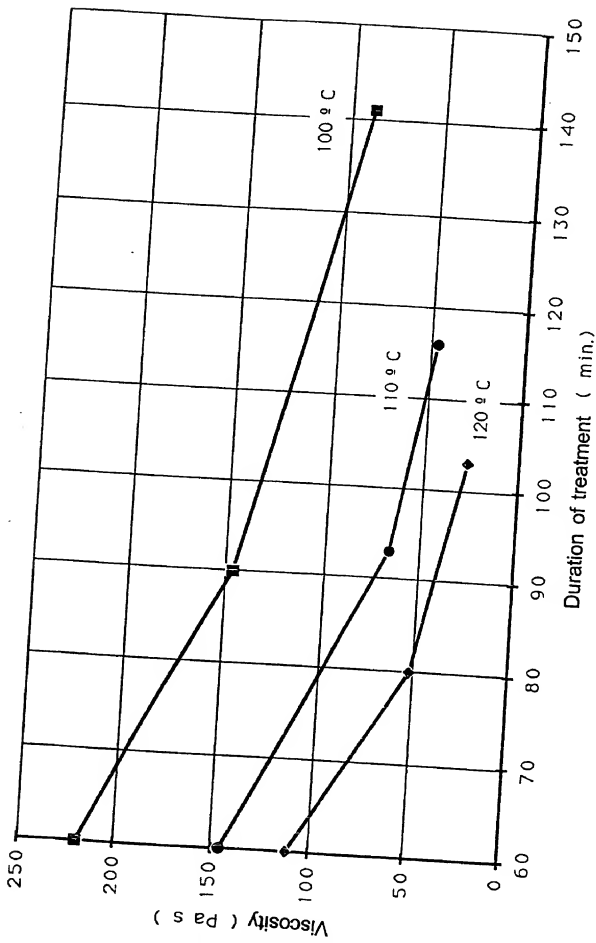
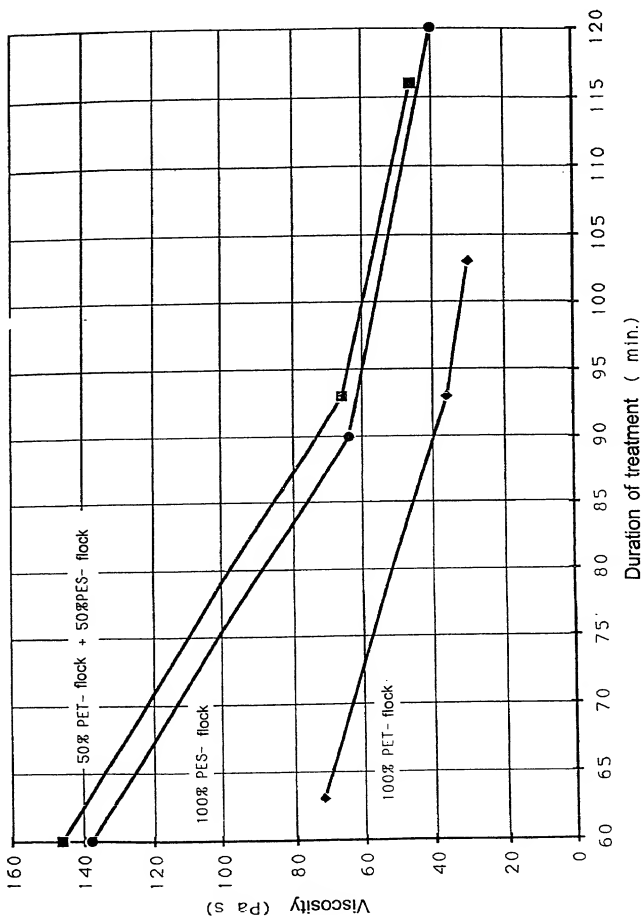


Figure 2

- 3 / 3 -

100280-246E1660

Fig. 3



2/PRTS - 1 -

09/913947

JC05 Rec'd PCT/PTO 2 0 AUG 2001

Process and device for producing elastane fibres from recycling material

This invention relates to a process and a device for producing elastane fibres from spinning solutions by a dry or wet spinning process using elastane recycling material, which optionally can be supplemented by admixing fresh solutions of elastane material in any proportions.

Elastane fibres are currently produced mainly by two fundamentally different processes, the dry spinning process and the wet spinning process. In the dry spinning process, the spinning solvent of a polyurethane solution, typically in dimethylacetamide, is removed from the fibres spun in the spinning cabinet; the removal is effected by hot gas in the spinning cabinet and heating the spinning cabinet. In the wet spinning process for producing elastane fibres, the elastane solution to be spun is usually degassed, then prefiltered and transferred to a spinning tank. The polymer solution is subsequently filtered and, after optional further introduction of additives, the spinning solution is delivered to the spinnerets by means of metering pumps. This procedure is described, for example, by F. Fourné in *Chemiefaser/Textilindustrie* 44/96th Year, June 1994, page 365, or in US-A 3 526 689.

By elastane solutions are meant solutions of polyurethanes or polyurethane ureas, which are conventionally made up of rigid and flexible segments, in suitable solvents such as dimethylacetamide or dimethylformamide. Usually polyester diols or polyether diols, depending on the intended use, are incorporated as flexible segments into the polyurethanes (polyurethane ureas).

Fresh spinning solutions, which are prepared by various solution processes, are generally used for producing elastane fibres. A practicable solution process is described, for example, in the document US-A 3 428 711. Better solution processes have been disclosed by Offenlegungsschriften DE 43 27 805 and DE 42 22 772 A1. Whereas, in the case of DE 43 27 805, one starts from a flow of liquid reaction

FAS 13

materials, which are heated in a microwave cavity resonator in order to prepare an elastane solution, DE 42 22 772 describes a process for preparing elastane spinning solutions which have a stable viscosity and are low in gel by using a special multistep nozzle reactor. A diagram of the complicated preparation processes is reproduced in
5 Chemiefasern/Textilindustrie 44/96th Year, June 1994, page 393, in Figure 3.

Solution processes for elastane material in which the use of pure recycled material, i.e. for example, of already spun fibrous elastane material, are hitherto known only for a quite specific polymer composition.

10 According to the Canadian Patent CA 771086, spandex yarns, which are produced from metaxylylenediamine, a polyglycol and a diisocyanate, can be dissolved with a solvent, filtered and reprocessed to form elastane fibres by dry spinning through nozzles. For solution to be possible, the yarns must have a linear polymer structure. In
15 general, however, conventional elastane yarns have cross-linked structures with a network in the form of biuret and allophanate bonding and consequently cannot easily be brought into solution.

In another known process, according to the Japanese Offenlegungsschrift JP
20 56122836, solid pieces of elastane are dissolved in isopropyl alcohol together with chloroacetaldehyde and a strong acid such as, for example, sulfuric acid and the solutions formed are further used for the production of synthetic leather. A use for elastane fibres is not described. The document DD 143916 describes the preparation of polyurethane-polyurea solutions by addition of recycled polyurethane solution during
25 the chain extending reaction. Solutions of that kind, however, are suitable only for synthetic leather production and not for producing elastane fibres. Besides these methods of utilising materials, so-called material recycling, methods of reusing raw materials, so-called chemical recycling, are also known (cf. E. Weigand: Plastverarbeiter 1995 46 (2) pages 88-92). In chemical recycling, the polyurethane
30 material is converted back into diamine and polyether polyol, for example, by hydrolysis with superheated steam under pressure. Other possible ways of converting

polyurethanes are provided by hydrogenation, pyrolysis or high-temperature gasification.

5 The reason why hitherto no recycling process for normal elastane waste, for example, from yarn production, has been found lies in the fact that in all solution processes it is necessary to apply high solution temperatures, usually above 100°C, in order to bring the elastane waste into solution. At these temperatures, however, the viscosity of the solution increases so much that no further processing can be carried out.

10 As in any elastane production process, waste arises in the form of residual fibres, for example, during disturbances in the course of production, during the spinning into other types and titres or as a result of defective batches, and as the raw materials price of the polymer material is high, as a rule more than 5.00 DM/kg polymer, the reuse of elastane waste is of great interest from the economic angle. The removal of fibrous waste (whether by dumping or by combustion) is likewise very cost-intensive, so that
15 the provision of a process which renders possible the reprocessing of elastane waste is also very relevant from the environmental aspect.

The object is achieved by a process and a device for producing elastane fibres from
20 spinning solutions by a dry or wet spinning process using elastane recycling material, which optionally can be carried out by admixing fresh elastane spinning solutions in any weight ratio, by adding secondary aliphatic amine, preferably diethyl amine, to the solutions.

25 The invention provides a process for producing elastane fibres by the dry spinning process or the wet spinning process using spinning solutions of elastane recycling material, optionally with the addition of fresh elastane solution, characterised in that

a) the waste obtained from elastane material, in particular from elastane fibres, is
30 cut up, in particular to a cut length of at least 0.1 mm, preferably to a cut length

of at least 1 mm, particularly preferably to a cut length of 2 to 10 mm, most preferably to a cut length of 3 to 5 mm,

- 5 b) the cut elastane material and 0.1 to 2 wt.%, based on the content of solid elastane, of a secondary aliphatic amine are introduced into the spinning solvent,
- c) the mixture of cut elastane material, spinning solvent and secondary aliphatic amine is heated at a temperature of 60°C to 150°C, with homogenisation,
- 10 d) the resulting homogeneous elastane spinning solution is prefiltered,
- e) optionally with the addition of fresh elastane solution, the elastane spinning solution is mixed at 70°C to 140°C with a fresh elastane solution, preferably having the same solids concentration, in any mixing ratio and heating is continued,
- 15 f) the resulting spinning solution is cooled to a temperature of at not more than 70°C, preferably from 50°C to 70°C, and filtered once more,
- 20 g) the finished spinning solution is optionally remixed, for example, by stirring, degassed and subsequently spun into elastane fibres by the dry spinning process or the wet spinning process.
- 25 Preferably the elastane waste consists of both elastanes based on polyether and elastanes based on polyester, or of wastes of mixed polyether- and polyester-containing elastanes in any mixing ratio.

30 The secondary aliphatic amine used is in particular diethylamine (DEA), in a quantity preferably of 0.3 to 1 wt.%, in particular preferably of 0.5 to 0.8 wt.%, based on solid elastane.

The concentration of the finished spinning solution in step g) is from 22 to 45 wt.%, preferably from 30 to 40 wt.%.

- 5 Dimethylacetamide is preferably used as the spinning solvent.

In the case of a mixed spinning solution, the elastane spinning solution is mixed with a fresh elastane solution in step e) for a period of 5 to 60 minutes.

- 10 The total mixing time in steps b) and c) together is advantageously at least 10 minutes, preferably from 60 to 150 minutes, particularly preferably 90 to 120 minutes.

The solution temperature in step c) is advantageously from 80°C to 120°C.

- 15 In the case where a mixture of waste elastane solution and fresh elastane solution is used, it is particularly advantageous to add the secondary amine, in particular diethylamine, to the fresh elastane solution not in step b) but in step f) and in the form of a stock batch of secondary amine and fresh elastane solution.

- 20 The recycling spinning solution and the fresh solution are preferably mixed together in a static mixer, preferably at 70°C to 140°C, in particular at 100°C to 120°C, for a period of 5 to 30 minutes and the spinning solution is subsequently cooled to 50°C to 70°C.

- 25 The solids content of the recycling spinning solution in proportion to the solids concentration of the total spinning solution comprising recycling spinning solution and fresh elastane solution is preferably at least 10 wt.%, preferably at least 20 wt.%.

- 30 The spinning of the spinning solutions by the wet spinning process is carried out more effectively in particular from spinnerets having a nozzle hole diameter of 0.10 to 0.3 mm, preferably of 0.1 to 0.2 mm.

The elastane material in step a) is introduced preferably in portions, in particular via a metering/weighing device, preferably at a rhythm of 1 to 10 kg/minute, particularly preferably 3 to 5 kg/minute, into the vortex created by an agitated disperser in the previously prepared spinning solution.

5

The invention also provides elastane fibres obtained from the process according to the invention, in particular with a titre of up to 10,000 dtex, their fibre strength being 0.5 to 0.95 cN/dtex, preferably 0.7 to 0.9 cN/dtex, and their elongation at tear being 500 to 750%, preferably 550 to 700%.

10

The invention further provides a device for carrying out the process according to the invention, consisting at least of a heatable mixing tank with an inlet for solid material, a mixer and a dispersing unit, of a mixing zone with static mixing elements, connected downstream of the mixing tank, of a cooling zone with mixing elements and of a filtering unit for subsequently filtering the prepared elastane spinning solution.

15

The dispersing unit consists in particular of at least one, particularly two, agitated dispersers.

20

The agitated dispersers are preferably equipped with screw-type segmented appliances.

The screw-type segmented appliances are selected in particular from suction cutters, webbed rings, kneading spirals and multiple current appliances.

25

The mixer in the mixing tank is preferably an anchor mixer and the mixing tank is provided with an additional cleaning device for the internal fittings of the mixer.

30

Surprisingly, it has been found that cutting up elastane bobbin material and wool from the spinning cabinets into short fibres of in particular approximately 3 to 5 mm cut length in a cutting mill with suitable tools causes no difficulties. These short fibres, which have a density of approximately 0.2 g/cm³, can be conveyed flawlessly over

00543947.002004

spirals and screws without sticking and clogging, for example, from a silo into the mixing tank which is used for the preparation of the spinning solution.

Special mixing units are particularly helpful in the preparation of suitable spinning solutions from waste fibrous material. Experiments in a single-shaft agitated tank with various stirrer attachments showed that satisfactory results could not be obtained because of very long dissolving times and mixing times and very high specific driving powers owing to the high viscosity of the mixed material. It also became apparent that, in the agitated tank already containing spinning solvent (DMAC), with increasing addition of short elastane waste fibres the viscosity can be controlled and decreased only if secondary aliphatic amines such as, for example, diethylamine (DEA) are added to the spinning solution in the above-mentioned quantities of 0.5 wt.% DEA, based on solid elastane. Without the addition of secondary aliphatic amines, dissolving, mixing and heating lead to spinning solutions whose viscosity rapidly increased. Such spinning solutions can no longer be controlled technically. These spinning solutions are so highly viscous that they tend to gel and, owing to the high pressures required, they are also no longer conveyable. The required viscosity for the spinning process can then be adjusted, depending on the contact time, by heating, for example, the diethylamine-containing spinning solutions to a temperature of up to 150°C. The adjustment of the viscosity of highly concentrated elastane solutions by reacting the solution with secondary aliphatic amines in order to produce spinning solutions for the dry or wet spinning of elastane fibres has already been described in DE 44 46 339. It is surprising that secondary amines can now also be used for the direct working up of elastane material.

As already mentioned above, single-shaft agitated tanks with various stirrer attachments, even with the application of secondary aliphatic amines to the spinning solvent, alone do not suffice to prepare suitable spinning solutions from waste elastane fibres. Time and again this leads to sticking to the edge of the agitated tank and to clogging inside the agitated tank. Stated in other words, inhomogeneous spinning

solutions which cannot be spun flawlessly into fibres are obtained, despite the addition of amine.

5 Unexpectedly, it has been found that these problems can be solved if one starts with mixing tanks having specific properties in order to prepare the spinning solutions.

10 A most particularly suitable mixing and dispersing machine has proved to be a unit which is equipped with double mantle for heating and cooling and at least one, preferably two, mixing shafts with dissolvers as well as a mixing shaft with anchor mixer (cf. Figure 1). The mixing shafts are advantageously fitted with screw-type segmented appliances such as, for example, suction cutters, webbed rings, kneading spirals or multiple current appliances. To prevent sticky deposits of polymer on the inside wall of the tank which is in contact with the product, the anchor mixer is preferably equipped with spring-mounted cleaning segments.

15 By means of such a mixing and dispersing machine, by initially using spinning solvent (in particular dimethylacetamide) and secondary aliphatic amine and by adding short waste fibres, it is possible successfully to prepare elastane spinning solutions having solids contents of up to 40 wt.% which can subsequently be spun flawlessly into
20 elastane fibres having good fibre properties.

It has proved to be advantageous to add the waste fibres via a metering/weighing device at a constant timed rhythm, in order to achieve a homogeneous solution process.
25

Furthermore, it has been found that it is particularly beneficial to introduce the short waste fibres as far as possible in the vicinity of the liquid vortices caused by the mixing shafts, in order to achieve a good wetting and solution of the fibres.

30 The solution process described is suitable for preparing spinning solutions which can be spun into elastane fibres by both the dry and the wet spinning process. The required

09913947.082001

spinning viscosity, which is distinctly higher in the dry spinning process than in a wet spinning process, can be adjusted via the amine content, the temperature of the solution and the residence time in the heater (cf. Figures 2 and 3).

- 5 In addition to spinning solutions from pure recycling material, mixtures with freshly prepared elastane spinning solutions can also be prepared in any mixing ratio, as described above.

10 The nature of the spinning solutions, whether they are, for example, spinning solutions which are by dissolving polyester- or polyether-containing fibrous material, or mixtures thereof in any composition, is irrelevant here (cf. Table 1). In all cases elastane fibres having good fibre qualities can be produced from such spinning solutions.

15 The elastane waste material (for example, fibres or wool) can contain up to 15 wt.% of conventional agents for fibre preparation, in particular, mineral oils or oils based on polyalkylsiloxane, for example, polydimethylsiloxane PDMS and/or ethoxylated PDMS or metal salts of fatty acids (for example, Mg stearate) or hydrotalcites alone or in any mixture.

20 The process using mixtures of fresh solutions and recycling material is particularly suitable for a continuous operation. The use of pure recycling solutions is particularly appropriate for a batch operation.

25 Elastane spinning solutions having a solids content of up to 40 wt.% can easily be prepared using the devices and solution processes described (cf. Example 11, Table I).

Besides the preparation of the spinning solution, the quality of the spinning solution is also decisive in the recycling process according to the invention. Good qualities in the spinning solution lead to readily filterable spinning solutions without rapid build-up of

30

pressure in the filtering units and to long nozzle lives and consequently to high product yields.

It has been found that the proposed solution process can be combined particularly advantageously with a wet spinning process with the use of spinnerets with nozzle hole diameters of 0.10 to 0.3 mm, in particular 0.1 to 0.2 mm, in order to achieve long nozzle lives. With finer nozzle hole diameters, for example, less than 0.10 mm, a considerably increased build-up of the pressure of the spinning solutions in front of the nozzle may possibly occur, which leads to distinctly shorter nozzle lives. Nozzle hole diameters of more than 0.3 mm have extremely low spraying speeds (less than 5) and possibly lead to very long delays in spinning (more than 50), which results in unstable spinning processes and broken fibres during fixing.

The spraying speed (S) is found from:

$$S = \frac{4 \cdot F \text{ (m/min)}}{Z \cdot d^2 \cdot \pi \cdot 100}$$

F = delivery (ccm/min)

Z = number of nozzle holes

d = nozzle hole diameter (cm)

The following Examples in association with Figures 1 to 3 serve to explain the invention further. All percentages given are based on the weight, unless indicated otherwise.

Figure 1 shows the structure of a device for preparing the elastane solution from waste fibres.

Figure 2 shows the decrease over time in the viscosity of a spinning solution in Example 1 at different treatment temperatures.

Figure 3 shows the decrease over time in the viscosity of different spinning solutions obtained from recycling material.

09943947.082004
T00280.746E1660

Examples

Example 1

5 a) Preparation of a spinning solution from recycling material

280 kg dimethylacetamide (DMAC) and 600 g diethylamine (DEA) were introduced into a 600 litre mixing tank 1 (cf. Figure 1) through an inlet 2. The two liquids were stirred and mixed together at room temperature by means of an anchor mixer 3. Then
10 the dissolver 4 (dispersing unit), which was equipped with screw-type segmented appliances 5 comprising suction cutter, dissolver discs and kneading spiral, was put into operation. The mantle heating of the mixing tank 1 was adjusted to 80°C. Subsequently, fibrous elastane material cut to lengths of 3 to 5 mm, originating from residual bobbins, was spirally conveyed alternately in portions at a rate of 4 kg/minute
15 via a balance (not shown) through the filler neck 6 into the mixer tank 1. The fibrous elastane material consisted of 60 kg fibrous elastane material containing polyether (PET) as the component of the flexible segments and of 60 kg fibrous elastane material containing polyester (PES) as the component of the flexible segments, which were introduced alternately via a balance, as mentioned above. The solids concentration was
20 30 wt.% and the proportion of diethylamine (DEA) was 0.5 wt.%, based on solid elastane.

The addition of the cut fibrous elastane material in portions was effected in such a way that the material was introduced into the vortex created by the dissolver 4, 5. The
25 dissolver 4, 5 was started at 700 revolutions per minute and after approximately 30 minutes, when the introduction of the fibres was complete, was operated at 1500 revolutions per minute for approximately 85 more minutes. The total stirring time was thus approximately 115 minutes.

30 In the course of this, the temperature of the mixture rose to approximately 110°C as a result of the mixing energy produced. The viscosity of the resulting spinning solution,

measured at 50°C, was 146 Pa.s after a stirring time of 60 minutes and 48 Pa.s at the end of the treatment period. The spinning solution was subsequently passed through a knot screen 13 and, together with the spinning solution b), was passed to a heated static mixer 7 (cf. Figure 1).

b) Preparation of the spinning solution from fresh solution having suitable wet-spinning viscosity and mixing with spinning solution a)

A 30 wt.% elastane spinning solution, which had been prepared as in Example 2 of the document DE-A 42 22 772 and had a spinning viscosity of 138 Pa.s (measured at 50°C), was placed in the agitated tank 8 (cf. Figure 1). (The content of DE-A 42 22 772 is hereby included with the present Application as a disclosure). In the side stream, a diethylamine-containing stock batch from a second agitated tank 9 was added to the spinning solution via a gear pump 10 and subsequently this spinning solution, together with the spinning solution a), was delivered by a further gear pump in the ratio 1:1 through a heated static mixer 7, and mixed. The static mixer 7, which was lined with mixing elements, was heated to 120°C. The residence time in the mixer was approximately 15 minutes. In the side stream, DEA-containing stock batch was added in a quantity such that the spinning solution b), prior to mixing and prior to combination with the spinning solution a), had a DEA-content of 0.5 wt.%, based on elastane solid. The stock batch in the agitated tank 9 had been prepared from 20 kg of 30 wt.% elastane spinning solution, as described above, by adding 4,800 g diethylamine (DEA) at 40°C, with stirring for 30 minutes. The combined spinning solutions from a) and b), after they had been passed through and treated in the mixer 7, were then cooled to 70°C in the second mixer 11, filtered through a filter press 12 and recirculated into a spinning tank with mixer (not shown). The combined elastane spinning solution, which consisted of 50% fresh solution (spinning solution b)), was visually homogeneous and contained neither large gel particles nor remains of undissolved fibrous material. The spinning viscosity of the solution, measured at 50°C, was 18 Pa.s.

c) Production of elastane fibres

The spinning solution from the spinning tank was passed directly to four 172-hole nozzles with 0.2 mm hole diameter, which were dipped into an aqueous spinning bath containing 22% DMAC. The filaments thus formed were drawn off at 80 m/min, coalesced, washed, fixed, prepared and wound up on a spooler at 125 m/min. The resulting fibres, with an individual titre of 751 dtex, had a fibre strength of 0.95 cN/dtex and an elongation at tear of 633%.

- Figure 2 shows the decrease in the viscosity of the spinning solution having the composition described in Example 1 at three different treatment temperatures in relation to the duration of treatment in the mixing tank 1 (cf. Figure 1). As can be seen from the curves, the viscosity can be adjusted within a wide range. Figure 3 shows the decrease in the viscosity of 30 wt.% spinning solutions obtained from recycling material of various compositions in relation to the duration of treatment. As the curves show, for the same duration of treatment in the mixing tank 1, under the same mixing conditions in the dissolver 4, 5 and at a mixing temperature of approximately 110°C, the spinning solution having the lowest viscosity is obtained from recycling spinning solutions containing pure fibrous elastane material based on polyether (PET) and the spinning solution having the highest viscosity is obtained from recycling spinning solutions consisting of fibrous elastane material containing polyether (PET) and polyester (PES) in the mixing ratio 1:1.

Example 2**a) Preparation of a spinning solution from pure recycling material**

5 280 kg DMAC and 600 g DEA were introduced into a 600 litre mixing tank 1 (cf. Figure 1) through an inlet 2. The two liquids mixed together at room temperature by means of stirring. Then the dissolver 4, which was equipped with screw-type segmented appliances 5, was started at 700 revolutions per minute. The mantle heating of the mixing tank 1 was adjusted to approximately 80°C. Subsequently, fibrous elastane material cut to lengths of 3 to 5 mm, originating from residual bobbins, was spirally conveyed in portions at a rate of 4 kg/minute via a balance through the filler neck 6 into the mixer tank 1. The fibrous elastane material consisted of 120 kg cut composite fibres containing polyether (PET) as the component of the flexible segments in the elastane. The solids concentration was again 30 wt.% and the proportion of DEA was 0.5 wt.%, based on solid. The cut composite fibres were again introduced into the vortex created by the dissolver 4. After approximately 30 minutes, when the introduction of the fibres was complete, the dissolver 4, was operated at 1500 revolutions per minute for a further 70 minutes. The total stirring time was thus approximately 100 minutes. In the course of this, the product temperature rose to approximately 110°C as a result of the mixing energy produced. The viscosity of the spinning solution, measured at 50°C, was 72 Pa.s after a stirring time of 63 minutes and 30 Pa.s at the end of the treatment period (cf. Figure 3). The spinning solution was subsequently passed through a knot screen 13 and then passed to a heated mixer 7. The static mixer 7, which was lined with mixing elements, was heated to 80°C. The residence time in the mixer 7 was approximately 10 minutes. The spinning solution in the mixer 11 was then cooled to 70°C, filtered through a filter press 12 and recirculated into a spinning tank (not shown). The spinning solution, which consisted of 100% recycling material, was visually again completely homogeneous and contained neither large gel particles nor remains of undissolved fibrous material. The spinning viscosity of the solution, measured at 50°C, was 20 Pa.s.

b) Production of elastane fibres

Subsequently the spinning solution from the spinning tank was in turn passed, as described in Example 1, directly to four 172-hole nozzles with a hole diameter of 0.25 mm instead of 0.2 mm, which dipped into an aqueous spinning bath containing 25% DMAC, then spun and aftertreated in the manner described in Example 1. The resulting elastane fibres had a titre of 822 dtex, a fibre strength of 0.79 cN/dtex and an elongation at tear of 626%.

10 Examples 3 to 12

Other compositions of recycling materials of varying chemical origin and fresh solutions of varying origin in different mixing ratios, corresponding to Examples 3 to 11, are listed in Table 1. There the abbreviation PES used indicates elastane spinning solutions containing a polyester component as the flexible segment, prepared as in Example 4 of DE 42 22 722 A1 and the abbreviation PET denotes elastane spinning solutions containing polyether material corresponding to Example 2 from DE 42 222 722 A1. All the spinning solutions were prepared as stated in Example No. 1 and spun into elastane fibres having a titre of 800 dtex. In the case of Examples 10 and 11, where more highly concentrated spinning solutions with a spinning concentration of 36 wt.% and 40.3 wt.% respectively were prepared, the concurrent heating in the mixing tank 1 was carried out at 110°C instead of 80°C. The total stirring time was increased to 130 minutes and the dissolver 4 was operated at 1750 instead of 1500 revolutions per minute. The product temperature was approximately 133°C and the viscosity of the spinning solutions at the end of the treatment time was 52 Pa.s and 46 Pa.s respectively. Finally, Example 12 is a Comparison Example which starts with a fresh pure solution of polyether. As can be seen from Table 1, both with 100% recycling spinning solution (cf. Example 3) and with all the mixing ratios, spinning of the solutions produced elastane fibres having a fibre strength of at least 0.72 cN/dtex and a fibre elongation at tear of at least 590%.

Table 1

Ex. No.	Composition of the spinning solution			Data on fibres		
	Fresh solution/ Type (%)	Recycling solution/ Type (%)	Spinning solution concn. (%)	Titre (dtex)	Strength (cN/dtex)	Elongation (%)
3	-	100/ PES	30	802	0.75	631
4	75 / PES	25 / PET	30	777	0.81	617
5	50 / PES	50 / PET	30	772	0.77	615
6	50 / PET	50 / PET	30	753	0.79	627
7	50 / PET	50 / PES	30	779	0.9	612
8	70 / PET	15 / PET; 15 / PES	30	817	0.76	590
9	75 / PET	25 / PES	30	814	0.73	649
10	50 / PET	50 / PET	36	797	0.78	624
11	50 / PET	50 / PET	40.3	808	0.72	663
12	100/ PET	Comparison	30	774	0.92	584

T00280" 246E1660

Claims

1. Process for producing elastane fibres by the dry spinning process or the wet spinning process using spinning solutions of elastane recycling material, optionally with the addition of fresh elastane solution, characterised in that
- a) the waste obtained from elastane material, in particular from elastane fibres, is cut up, in particular to a cut length of at least 0.1 mm, preferably to a cut length of at least 1 mm, particularly preferably to a cut length of 2 to 10 mm, most preferably to a cut length of 3 to 5 mm,
- b) the cut elastane material and 0.1 to 2 wt.%, based on the content of solid elastane, of a secondary aliphatic amine are introduced into the spinning solvent,
- c) the mixture of cut elastane material, spinning solvent and secondary aliphatic amine is heated at a temperature of 60°C to 150°C, with homogenisation,
- d) the resulting homogeneous elastane spinning solution is prefiltered,
- e) optionally with the addition of fresh elastane solution, the elastane spinning solution is mixed at 70°C to 140°C with a fresh elastane solution, preferably having the same solids concentration, in any mixing ratio and heating is continued,
- f) the resulting spinning solution is cooled to a temperature of at not more than 70°C, preferably from 50°C to 70°C, and filtered once more,

g) the finished spinning solution is optionally remixed, for example, by stirring, degassed and subsequently spun into elastane fibres by the dry spinning process or the wet spinning process.

- 5 2. Process according to claim 1, characterised in that the elastane waste consists of both elastanes based on polyether and elastanes based on polyester, or of wastes of mixed polyether- and polyester-containing elastanes in any mixing ratio.
- 10 3. Process according to claim 1 or 2, characterised in that the secondary aliphatic amine used is diethylamine (DEA), in a quantity preferably of 0.3 to 1 wt.%, in particular preferably of 0.5 to 0.8 wt.%, based on solid elastane.
- 15 4. Process according to claims 1 to 3, characterised in that the concentration of the finished spinning solution in step g) is from 22 to 45 wt.%, preferably from 30 to 40 wt.%.
- 20 5. Process according to claims 1 to 4, characterised in that dimethylacetamide is used as the spinning solvent.
6. Process according to claims 1 to 5, characterised in that the elastane spinning solution is mixed with a fresh elastane solution in step e) for a period of 5 to 60 minutes.
- 25 7. Process according to claims 1 to 6, characterised in that the total mixing time in steps b) and c) together is at least 10 minutes, preferably from 60 to 150 minutes, particularly preferably 90 to 120 minutes.
- 30 8. Process according to claims 1 to 7, characterised in that the solution temperature in step c) is from 80°C to 120°C.

09513947.082004

9. Process according to claims 1 to 8, characterised in that in the case where a mixture of waste elastane solution and fresh elastane solution is used, the required addition of secondary amine, in particular of diethylamine, to the fresh elastane solution is carried out not in step b) but in step e) and in the form of a stock batch of secondary amine and fresh elastane solution.
10. Process according to claims 1 to 9, characterised in that the recycling spinning solution and the fresh solution are preferably mixed together in a static mixer, preferably at 70°C to 140°C, in particular at 100°C to 120°C, for a period of 5 to 30 minutes and the spinning solution is subsequently cooled to 50°C to 70°C.
11. Process according to claims 1 to 10, characterised in that the solids content of the recycling spinning solution in proportion to the solids concentration of the total spinning solution comprising recycling spinning solution and fresh elastane solution is at least 10 wt.%, preferably at least 20 wt.%.
12. Process according to claims 1 to 11, characterised in that the spinning of the spinning solutions by the wet spinning process is effected from spinnerets having a nozzle hole diameter of 0.10 to 0.3 mm, preferably of 0.1 to 0.2 mm.
13. Process according to claims 1 to 12, characterised in that the elastane material in step a) is introduced in portions, in particular via a metering/weighing device, preferably at a rhythm of 1 to 10 kg/minute, particularly preferably 3 to 5 kg/minute, into the vortex created by an agitated disperser in the previously prepared spinning solvent.
14. Elastane fibres obtained from a process according to one of claims 1 to 13, in particular with a titre of up to 10,000 dtex, characterised in that their fibre strength is 0.5 to 0.95 cN/dtex, preferably 0.7 to 0.9 cN/dtex, and the elongation at tear is 500 to 750%, preferably 550 to 700%.

15. Device for carrying out the process according to one of claims 1 to 13, consisting at least of a heatable mixing tank 1 with an inlet 2, 6 for solid material, a mixer 3 and a dispersing unit 4, 5, of a mixing zone 7 with static mixing elements, connected downstream of the mixing tank 1, of a cooling zone 11 with mixing elements and of a filtering unit 12 for subsequently filtering the prepared elastane spinning solution.
16. Device according to claim 15, characterised in that the dispersing unit 4, 5, consists of at least one, in particular two, agitated dispersers.
17. Device according to claim 16, characterised in that the agitated disperser(s) 4 is/are equipped with screw-type segmented appliances 5.
18. Device according to claim 16, characterised in that the agitated disperser(s) 4 is/are equipped with screw-type segmented appliances 5 selected from suction cutters, webbed rings, kneading spirals and multiple current appliances.
19. Device according to one of claims 14 to 17, characterised in that the mixer 3 is an anchor mixer and the mixing tank 1 is provided with an additional cleaning device for the internal fittings of the mixer 3.

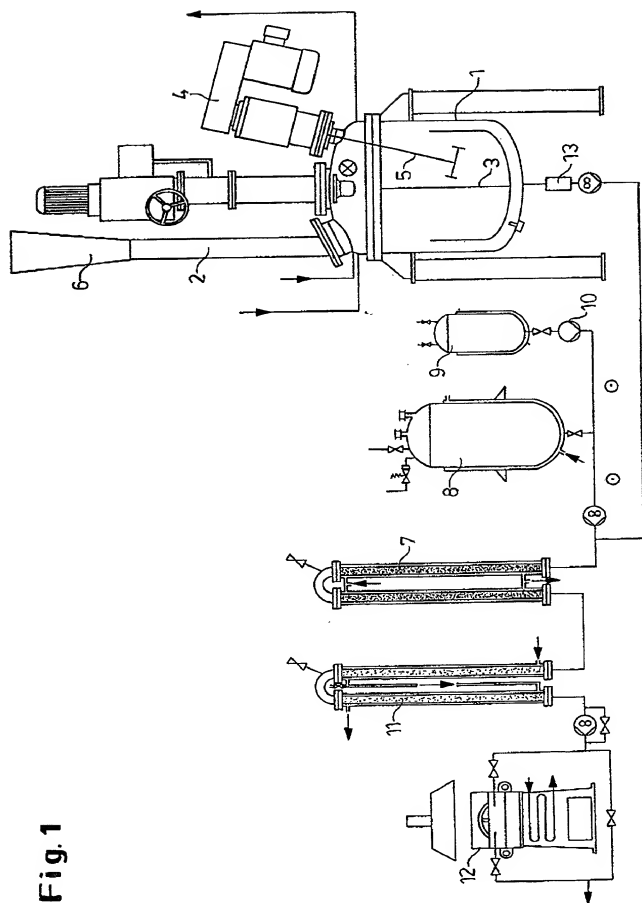


Fig. 1

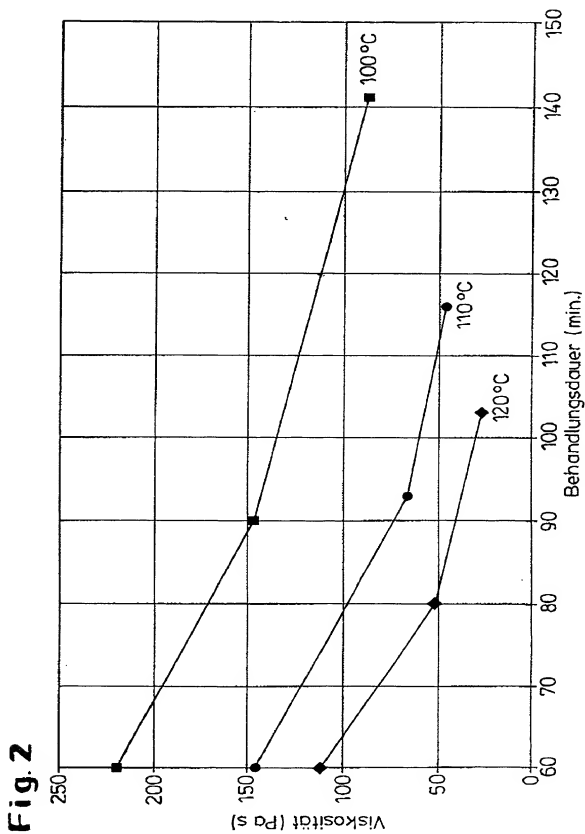
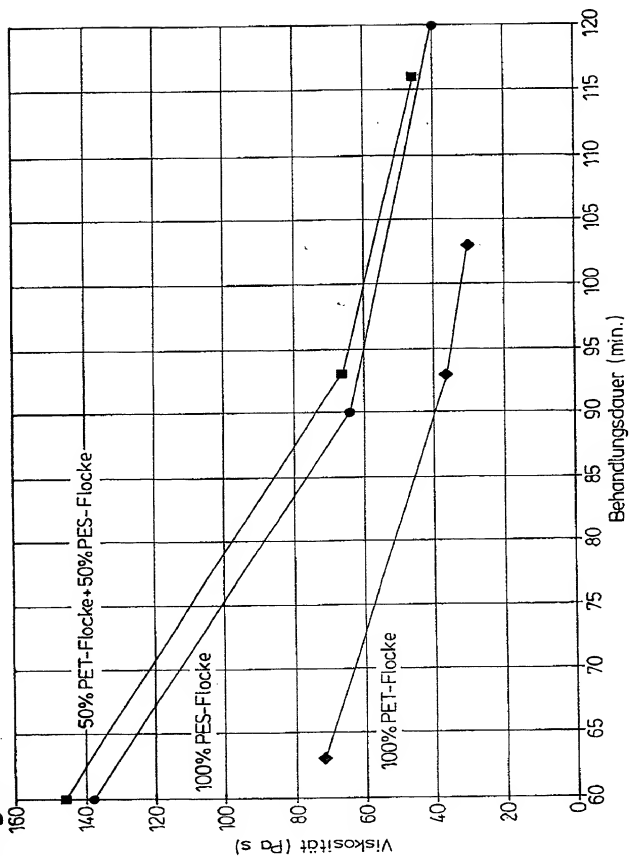


Fig. 3



COMBINED DECLARATION AND POWER OF ATTORNEY

ATTORNEY DOCKET NO

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

METHOD AND DEVICE FOR PRODUCING ELASTANE THREADS FROM RECYCLING MATERIAL

the specification of which is attached hereto,

or was filed on **February 14, 2000**

as a PCT Application Serial No. **PCT/EP00/01206**

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the patentability of this application in accordance with Title 37, Code of Federal Regulations, §1.56.

I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s), the priority(ies) of which is/are to be claimed:

199 07 830.0
(Number)

Germany
(Country)

February 24, 1999
(Month/Day/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose the material information as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

(Application Serial No.)

(Filing Date)

(Status)

(patented, pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FAS 13-US

HG

000280.2463666

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorneys and/or agents to prosecute this application and transact all business in the Patent and Trademark Office connected therewith:

Kurt G. Briscoe, Reg. No. 33,141; William C. Gerstenzang, Reg. No. 27,552 and Stephen G. Ryan, Reg. No. 39,015, all of 220 East 42nd Street, 30th Floor, New York, New York 10017, and William R. Robinson, Reg. No. 27,224, Dary E. Zoneraich, Reg. No. 37,267 and Mark A. Montana, Reg. No. 44,948, all of 721 Route 202-206, Bridgewater, New Jersey 08807, my attorneys with full power of substitution and revocation

Send Correspondence To:
NORRIS, McLAUGHLIN & MARCUS

220 East 42nd Street, 30th Floor
New York, New York 10017

Direct Telephone Calls To:

(212) 808-0700

FULL NAME OF SOLE OR FIRST INVENTOR <u>Ulrich Reinehr</u>		INVENTOR'S SIGNATURE <i>Ulrich Reinehr</i>	DATE <u>01.08.01</u>
RESIDENCE <u>D 41539 Dormagen, Germany DEX</u>		CITIZENSHIP <u>German</u>	
POST OFFICE ADDRESS <u>Röntgenstr. 29, D 41539 Dormagen, Germany</u>			
FULL NAME OF SECOND INVENTOR <u>Tilo Sehm</u>		INVENTOR'S SIGNATURE <i>Tilo Sehm</i>	DATE <u>20.7.01</u>
RESIDENCE <u>D 40591 Düsseldorf, Germany DEX</u>		CITIZENSHIP <u>German</u>	
POST OFFICE ADDRESS <u>Celsiusweg 18, D 40591 Düsseldorf, Germany</u>			
FULL NAME OF THIRD INVENTOR <u>Wolfgang Anderheggen</u>		INVENTOR'S SIGNATURE <i>Wolfgang Anderheggen</i>	DATE <u>25.07.01</u>
RESIDENCE <u>D 41539 Dormagen, Germany DEX</u>		CITIZENSHIP <u>German</u>	
POST OFFICE ADDRESS <u>Kneippstr. 17, D 41539 Dormagen, Germany</u>			
FULL NAME OF FOURTH INVENTOR <u>Toni Herbertz</u>		INVENTOR'S SIGNATURE <i>Toni Herbertz</i>	DATE <u>24.7.01</u>
RESIDENCE <u>D 41540 Dormagen, Germany DEX</u>		CITIZENSHIP <u>German</u>	
POST OFFICE ADDRESS <u>Hauptstr. 79, D 41540 Dormagen, Germany</u>			
FULL NAME OF FIFTH INVENTOR <u>Helmut Judat</u>		INVENTOR'S SIGNATURE <i>Helmut Judat</i>	DATE <u>05.08.01</u>
RESIDENCE <u>D 40764 Langenfeld, Germany DEX</u>		CITIZENSHIP <u>German</u>	
POST OFFICE ADDRESS <u>Oskar-Erbs16h-Str. 44a, D 40764 Langenfeld, Germany</u>			
FULL NAME OF SIXTH INVENTOR		INVENTOR'S SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF SEVENTH INVENTOR		INVENTOR'S SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			
FULL NAME OF EIGHTH INVENTOR		INVENTOR'S SIGNATURE	DATE
RESIDENCE		CITIZENSHIP	
POST OFFICE ADDRESS			